

Influence of different vehicles on the pH of calcium hydroxide pastes

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Abstract: The main known benefit of calcium hydroxide as an intracanal medicament lies in the bactericidal effect conferred by its pH. The objective of this work was to determine the influence of the vehicle on the pH of calcium hydroxide pastes after usage in patients and *in vitro*. The incisor root canals of 180 patients were instrumented and filled with calcium hydroxide pastes containing distilled water, chlorhexidine, propylene glycol, anesthetic solution, camphorated *p*-monochlorophenol and camphorated *p*-monochlorophenol-propylene glycol. The pH of the paste in the patients' root canals was measured at 7, 14 and 21 days. Similarly, pH was measured *in vitro* up to 21 days. The pH of all the pastes remained constant throughout the time periods assessed. The calcium hydroxide-water combination showed significantly higher pH values than the other pastes in clinical use. Comparative analysis showed that the pH values of the anesthetic solution, camphorated *p*-monochlorophenol and camphorated *p*-monochlorophenol-propylene glycol were significantly higher *in vitro*. The type of vehicle was shown to influence the final pH of the pastes. However, the alkalinity of all pastes was maintained over time under the experimental conditions. (*J. Oral Sci.* 46, 107-111, 2004)

Key words: calcium hydroxide; pH; endodontic vehicles.

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Introduction

Calcium hydroxide (Ca(OH)₂), widely used for apexification and pulp capping procedures (1), is nowadays widely used as an intracanal medicament in endodontic therapy (2,3).

Clinically, Ca(OH)₂ appears to control infection and to reduce the incidence of symptoms between appointments (4,5). This antibacterial effect is believed to be dependent on its strongly alkaline pH (12.4) which is maintained over a long period of time because of the low solubility of the compound.

Tronstad et al. (6) suggested that Ca(OH)₂ placed in the root canal elevates the pH, producing an alkaline environment in areas of root resorption by diffusion of hydroxyl ions (OH⁻) through the dentinal tubules. High pH is bactericidal and also inhibits osteoclastic activity. Foster et al. (7) demonstrated that removal of the intracanal smear layer may facilitate Ca(OH)₂ diffusion. Nerwich et al. (8) demonstrated that hydroxyl ions derived from a Ca(OH)₂ dressing diffuse through root dentin faster and reach higher levels in the cervical part of the root than in the apical region.

Ca(OH)₂ powder for root canal dressing has been mixed with different vehicles such as distilled water, camphorated monochlorophenol (CMCP), normal saline, cresatin, glycerin and propylene glycol (PG) (1). The dissociation of Ca(OH)₂ into OH⁻ and Ca²⁺ depends on the vehicle used to prepare the paste. Simon et al. (9) demonstrated that the vehicle can exert a great influence on the release of ions. Safavi and Nakayama (10) concluded that high concentrations of glycerin and PG as mixing vehicles may decrease the effectiveness of Ca(OH)₂ as a root canal dressing. Other authors (11) demonstrated that Ca(OH)₂/CMCP/glycerin paste rapidly kills bacteria and

indicated that the CMCP increased the antimicrobial effect of the $\text{Ca}(\text{OH})_2$ paste. Sjögren et al. (12) demonstrated *in vivo* that $\text{Ca}(\text{OH})_2$ dressings efficiently eliminate bacteria which may survive biomechanical instrumentation, and that reliable and predictable results can be achieved by dressing the canal with $\text{Ca}(\text{OH})_2$ for 7 days. However, few studies have investigated the conservation of pH in $\text{Ca}(\text{OH})_2$ *in vivo*. The aim of the present study was to determine the influence of vehicles on the pH of $\text{Ca}(\text{OH})_2$ pastes after usage in patients and *in vitro*.

Materials and Methods

In vitro study

$\text{Ca}(\text{OH})_2$ pastes were prepared by adding to $\text{Ca}(\text{OH})_2$ powder (Anedra Lab., Buenos Aires, Argentina) the following vehicles: distilled water, 0.2% chlorhexidine gluconate (ICN Biomedicals Inc, Ohio, USA), 99.5% PG (Anedra Lab., Buenos Aires, Argentina), 4% articaine chlorhydrate (anesthetic solution; Totalcaina Forte, Microsules-Bernabó S.A., Lab., Buenos Aires, Argentina), 11.8% CMCP (Farmadental Lab., Buenos Aires, Argentina) and 11.8% CMCP-99.5% PG. The concentrations of the vehicles were evaluated quantitatively (13).

Using these pastes, aqueous solutions of $\text{Ca}(\text{OH})_2$ to a final concentration of 0.1 M were prepared in order to measure pH. These solutions were stored at 37°C in sterile tubes. The pH was measured at 0, 1, 7, 14 and 21 days in triplicate.

Clinical study

A total of 180 maxillary incisors with pulp necrosis and radiographically visible chronic periapical lesions were selected. Patients of both sexes aged from 20 to 50 years and who attended the Endodontics Department of the Faculty of Odontology of the National University of Tucumán were considered. Informed consent was obtained from all the subjects who participated.

After isolation with a rubber dam, carious tissue was removed from the teeth with a carver and a slow-speed handpiece. The pulp tissue remaining in the canal was removed with K-files. The working length of the roots was determined by inserting a 15 K-file and radiographically monitoring the process. The canals were instrumented with a step back technique (14) up to a 45 or 50 master apical file. After using each instrument, the canals were irrigated with 2 ml of 1% sodium hypochlorite. All teeth were cleaned and shaped to receive a temporary paste filling. Teeth were filled using the last K-file employed in the canal preparation, aided by absorbent paper points and vertical pluggers (15). The access cavities were closed with Cavit (Espe, Seefeld, Germany) and glass ionomer

restorative cement (Fuyi II, GC Corp., Tokyo, Japan). The temporary pastes were retained in the root canal for periods of 7, 14 and 21 days.

Patients were randomly divided into six groups each containing 30 teeth. The paste fillings were prepared from $\text{Ca}(\text{OH})_2$ powder and the same vehicles employed in the *in vitro* study (distilled water, 0.2% chlorhexidine gluconate, 99.5% PG, 4% articaine chlorhydrate, 11.8% CMCP and 11.8% CMCP-99.5% PG).

At each time point, 10 pastes from each group were removed with K-files and collected in separate Eppendorf tubes previously weighed on a precision scale (Acculab LA-Series Analytical Balances, Newtown, Canada). The tubes with the pastes were re-weighed and the difference between the initial and final weights was calculated. In this way the weight of the extracted paste from the root canal was obtained. Then the pastes were dissolved with distilled water to a final $\text{Ca}(\text{OH})_2$ concentration of 0.1 M, according to the following formula: volume of distilled water added (ml) = weight of the paste (g) / 0.0741 (mMW of $\text{Ca}(\text{OH})_2$) × $\text{Ca}(\text{OH})_2$ concentration (0.1 M). The solutions were used to obtain pH measurements.

pH measurement

The pH was determined with a digital pH meter (Broadley-James Irvine, California, USA) for small volumes (sensitivity: 0.01 pH units), calibrated to pH 7 and 4 with standard buffer solutions before use. The pH was determined by placing the refillable calomel electrode in a 15- μl sample on a slide for 10 seconds. The electrode was washed with distilled water and wiped dry between readings.

Statistics

To determine the relationship between the vehicles and the storage periods, two-way analysis of variance (ANOVA) was applied. Where interference between the tested factors was registered, the one-way ANOVA test was performed. Dunnett's T3 multiple range test was used to determine differences among the vehicles and among the storage periods. Differences at $P < 0.05$ were considered statistically significant.

Differences between between clinical and *in vitro* pH determinations were statistically analyzed using Student's *t*-test.

Results

Figure 1 shows the *in vitro* results. All the samples maintained their pH throughout the tested time intervals. Statistical analysis showed interaction between the factors, so the one-way ANOVA test for the pastes and the storage

periods was applied separately. No significant difference in pH was found for the different time intervals. However, significant differences in pH were observed among the pastes tested. Dunnett's T3 multiple range test showed no significant differences among the $\text{Ca}(\text{OH})_2$ pastes with distilled water, chlorhexidine, PG and anesthetic solution, but statistically significant differences were observed between these pastes and those containing CMCP and CMCP-PG.

The pH values recorded for the six pastes tested in the clinical study are shown in Fig. 2. Results obtained by two-way ANOVA showed no interactions between pastes and storage periods. No statistically significant difference in pH was observed among the different time intervals, but statistically significant differences were observed among the different pastes tested. Dunnett's T3 test revealed that the paste containing distilled water was significantly different from the other pastes at all storage periods. The highest pH values corresponded to the paste with distilled water, followed by the pastes prepared with chlorhexidine, CMCP, PG, CMCP-PG and anesthetic solution. There was no significant difference between the pastes containing CMCP, PG, CMCP-PG and anesthetic solution. However, the $\text{Ca}(\text{OH})_2$ paste containing chlorhexidine was significantly different from the pastes containing distilled water, PG, CMCP-PG and anesthetic solution.

Figure 3 shows a comparison of the *in vitro* and clinical results at 7, 14 and 21 days. The pH values remained constant throughout the time intervals both in the *in vitro* and clinical studies. After all the storage periods $\text{Ca}(\text{OH})_2$

pastes prepared with CMCP-PG, PG and anesthetic solution *in vitro* showed significantly higher pH values than in the clinical study. However, no significant differences in pastes with distilled water, chlorhexidine and CMCP were recorded.

Discussion

Since the introduction of $\text{Ca}(\text{OH})_2$ for use in dentistry by Herman in 1920, this medicament has been reported to promote healing in many clinical situations. The properties of $\text{Ca}(\text{OH})_2$ have been investigated by many authors, and clinical applications of this substance have been reported (1-4). Calcium release and an alkaline pH are extremely important for the biological and microbiological performance of the material for dental use (16).

In this study, no difference in the pH of each of the $\text{Ca}(\text{OH})_2$ pastes was observed over time; the tested pastes maintained their alkalinity even in an Eppendorf tube (*in vitro* study) as in the clinical study. *In vitro*, pastes with chlorhexidine, PG and anesthetic solution showed a similar pH to those containing distilled water; however, in the clinical experiment the pH of the paste containing distilled water was significantly different from the other pastes.

The pH value of the paste containing distilled water in patients was higher than in the *in vitro* study; however, no significant differences were observed at any time point. Of the remaining pastes, the pH values in the clinical study were lower than in the *in vitro* study, and the values for the pastes containing PG, anesthetic solution and

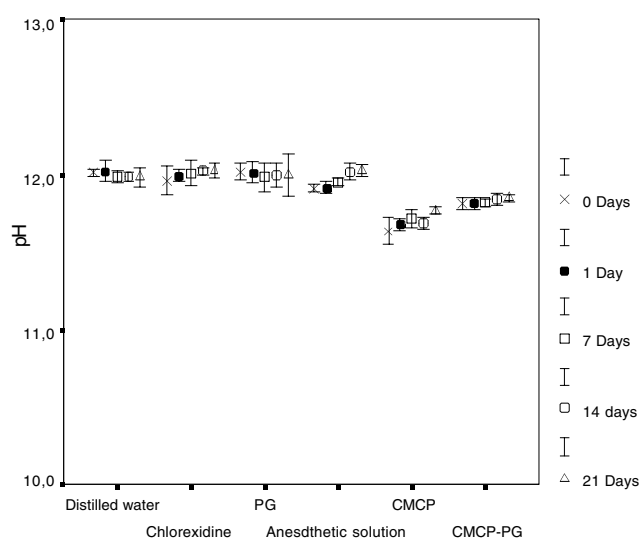


Fig. 1 Mean and standard error of pH values of 0.1 M $\text{Ca}(\text{OH})_2$ solutions prepared from $\text{Ca}(\text{OH})_2$ pastes added to different vehicles, evaluated over time in the *in vitro* experiment.

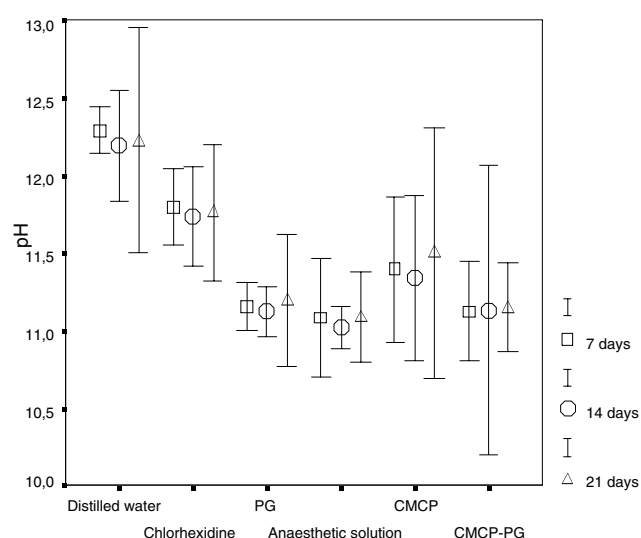
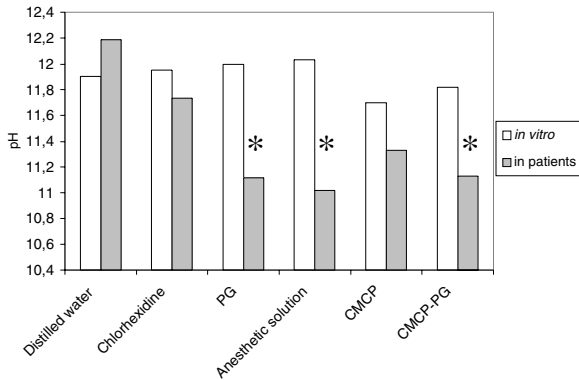
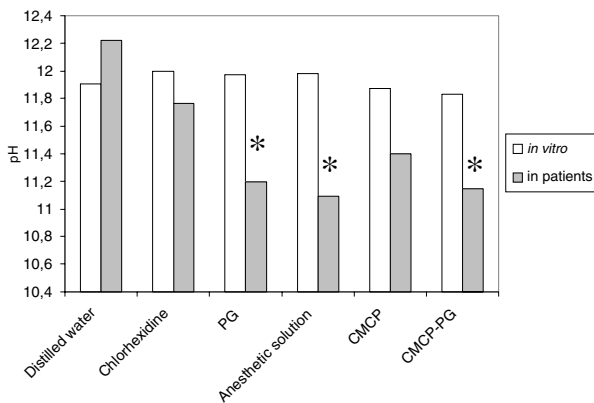


Fig. 2 Mean and standard error of pH values of 0.1 M $\text{Ca}(\text{OH})_2$ solutions prepared from $\text{Ca}(\text{OH})_2$ pastes added to different vehicles, evaluated over time in the clinical experiment.

a



b



c

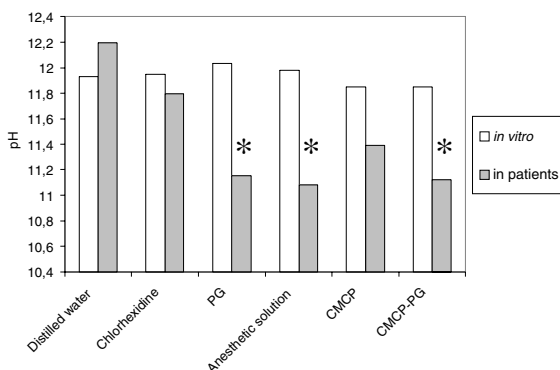


Fig. 3 Comparison between the *in vitro* and the clinical pH values of 0.1 M $\text{Ca}(\text{OH})_2$ solutions prepared from $\text{Ca}(\text{OH})_2$ pastes added to different vehicles, evaluated over time periods of a) 7 days, b) 14 days and c) 21 days.

CMCP-PG were found to be significantly different. In the *in vitro* experiment, aqueous solutions were used, providing optimal conditions for the release of OH^- ions. However, in the instrumented canal the release of OH^- ions could be limited by a decline in the availability of water molecules (12). In the *in vitro* study, $\text{Ca}(\text{OH})_2$ was mixed with PG to a final $\text{Ca}(\text{OH})_2$ concentration of 0.1 M and the aqueous solution was maintained for 14 days. In the clinical study, 99.5% PG was mixed with $\text{Ca}(\text{OH})_2$ powder in a ratio of 1/1 (13), and maintained in the root canal as a paste over the designated time period. The 0.1 M solution was prepared immediately before measurement of the pH. As reported by Safavi and Nakayama (10), the dissociation of electrolytes in solution can be estimated by measuring the solution conductivity, and high concentrations of PG reduce the conductivity of the $\text{Ca}(\text{OH})_2$ solution, suggesting that $\text{Ca}(\text{OH})_2$ does not dissociate in the presence of PG. However small amounts of PG in water result in increased conductivity of the $\text{Ca}(\text{OH})_2$ solution. In these experiments the factors considered by Safavi and Nakayama (10) would explain the different behavior observed in Fig. 3 between the *in vitro* and the clinical results. In the *in vitro* study it would be expected that conductivity would be increased and more dissociation of ions would occur.

Many reports claim that the vehicles mixed with $\text{Ca}(\text{OH})_2$ can influence the pH of the paste and the velocity of OH^- ion diffusion through the dentinal tubules (9,17). Esberard et al. (18) indicated that $\text{Ca}(\text{OH})_2$ in a CMCP vehicle diffuses more rapidly than $\text{Ca}(\text{OH})_2$ in an aqueous vehicle in the cervical and middle regions of the teeth. In the *in vitro* study, since pastes prepared with CMCP-PG showed a lower pH than in the clinical study, the proportion of PG may be responsible for the different pH values registered.

Differences were also observed in pastes containing anesthetic solution, which is an aqueous vehicle with an initial pH markedly lower than the pH of distilled water and chlorhexidine (13). In clinical situations the release of OH^- ions is not as controllable as in the *in vitro* situation because of the efficient buffering systems present in the tissues and also because of the influence of the acidic pH of the inflamed tissues. It is also possible that the buffering effect of the dentin would only occur in the paste-tissue interface.

Controversial data on the diffusion of OH^- ions and pH values have been reported. Tronstad et al. (6) found that the pH of dentin increased after placing $\text{Ca}(\text{OH})_2$ in the root canal. Esberard et al. (18) measured pH in external cavities and reported that non-setting $\text{Ca}(\text{OH})_2$ pastes maintained a high pH for at least 120 days. Çalt et al. (17) demonstrated that non-setting $\text{Ca}(\text{OH})_2$ pastes released Ca^{2+} through root dentin, but did not increase the pH of the

media, indicating that OH⁻ ions did not diffuse out the dentin.

In the present study, vehicles used to prepare the Ca(OH)₂ pastes were shown to influence the final pH of the pastes. Although some significant differences were found, both the *in vitro* and the clinical study recorded pH values between 11 and 12. The different vehicles permit OH⁻ ion release from Ca(OH)₂ pastes to different degrees, as mentioned by other authors (9). The pH of all the Ca(OH)₂ pastes both *in vitro* and clinically remained constant throughout the time intervals examined. According to this criterion, any of the vehicles tested are suitable for clinical use as an intracanal medicament.

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References

1. Fava LR, Saunders WP (1999) Calcium hydroxide pastes: classification and clinical indications. *Int Endod J* 32, 257-282
2. Siqueira JF Jr, de Uzeda M (1997) Intracanal medicaments: evaluation of the antibacterial effects of chlorhexidine, metronidazole and calcium hydroxide associated with three vehicles. *J Endod* 23, 167-169
3. Weigher R, Rosendahl R, Lost C (2000) Influence of calcium hydroxide intracanal dressing on the prognosis of teeth with endodontically induced periapical lesions. *Int Endod J* 33, 219-226
4. Siqueira JF Jr, Lopes HP (1999) Mechanisms of antimicrobial activity of calcium hydroxide: a critical review. *Int Endod J* 32, 361-369
5. Hammarström LE, Blomlöf LB, Feiglin B, Lindskog SF (1986) Effect of calcium hydroxide treatment on periodontal repair and root resorption. *Endod Dent Traumatol* 2, 184-189
6. Tronstad L, Andreasen JO, Hasselgren G, Kristerson L, Riis I (1981) pH changes in dental tissues after root canal filling with calcium hydroxide. *J Endod* 7, 17-21
7. Foster KH, Kulild JC, Weller RN (1993) Effect of smear layer removal on the diffusion of calcium hydroxide through radicular dentin. *J Endod* 19, 136-140
8. Nerwich A, Fidgor D, Messer HH (1993) pH changes in root dentin over a 4-week period following root canal dressing with calcium hydroxide. *J Endod* 19, 302-306
9. Simon ST, Bhat KS, Francis L (1995) Effect of four vehicles on the pH of calcium hydroxide and the release of calcium ion. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 80, 459-464
10. Safavi K, Nakayama TA (2000) Influence of mixing vehicle on dissociation of calcium hydroxide in solution. *J Endod* 26, 649-651
11. Siqueira Jr JF, de Uzeda M (1998) Influence of different vehicles on the antibacterial effects of calcium hydroxide. *J Endod* 24, 663-665
12. Sjögren U, Fidgor D, Spångberg L, Sundqvist G (1991) The antimicrobial effect of calcium hydroxide as short-term intracanal dressing. *Int Endod J* 24, 119-125
13. Pacios MG, de la Casa ML, Bulacio MA, López ME (2003) Calcium hydroxide's association with different vehicles: *in vitro* action on some dentinal components. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 96, 96-101
14. Leonardo MR, Leal JM (1994) Preparación biomecánica de conductos radiculares. In: *Endodoncia: Tratamiento de los conductos radiculares 1994*, 2nd ed. Leonardo MR, Leal JM eds, Edit. Médica Panamericana, Buenos Aires, Argentina, 296-320 (in Spanish)
15. Estrela C, Mamede Neto I, Lopes HP, Estrela CRA, Pécora JD (2002) Root canal filling with calcium hydroxide using different techniques. *Braz Dent J* 13, 53-56
16. Duarte MA, Demarchi AC, Giaxa MH, Kuga MC, Fraga SC, de Souza LC (2000) Evaluation of pH and calcium ion release of three root canal sealers. *J Endod* 26, 389-390
17. Çalt S, Serper A, Özçelik B, Dalat MD (1999) pH changes and calcium ion diffusion from calcium hydroxide dressing materials through root dentin. *J Endod* 25, 329-331
18. Esberard RM, Carnes DL Jr, del Rio CE (1996) Changes in pH at the dentin surface in roots obturated with calcium hydroxide pastes. *J Endod* 22, 402-405